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SINTERABILITY OF TUNGSTEN POWDER CVD COATED WITH NICKEL AND IRON

SHARON MULLIGAN and ROBERT J. DOWDING
MATERIALS PRODUCIBILITY BRANCH

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ABSTRACT

Three lots of tungsten powder coated with nickel and iron by chemical vapor deposition (CVD) were characterized as to its composition, particle size, and size distribution. This was accomplished through the use of the scanning electron microscope (SEM) with a digital X-ray dot mapping attachment, DC plasma emission, and Microtrac analysis. The sinterability of the powder was also evaluated through sintering trails at temperatures above and below the liquidus of the matrix phase. It was found that the coating of nickel and iron on the tungsten substrate was very uniform, but it was also difficult to obtain high sintered densities due to the near monosized nature of the powder. The highest sintered density obtained was 90% of the theoretical value. It was concluded that to obtain full density a wider distribution of the particle size would be needed.

CONTENTS

INTRODUCTION

Tungsten powder with a chemical vapor deposited (CVD) coating of nickel and iron was produced by Ultramet, Pacoima, CA, under the U. S. Army's Small Business Innovative Research (SBIR) Program.[1,2] This processing technique for tungsten powder is a part of the U. S. Army's effort to enhance the mechanical and ballistic properties of tungsten-based materials for use as kinetic energy penetrators.

This is a continuing project. Over a four month period, three lots of powder, each with a different composition, was received from Ultramet. The purpose was to characterize the powder resulting from the coating process, as well as to evaluate its sinterability. The powder was characterized by performing chemical analyses to determine the amount of each element present and by determining its particle size distribution. In order to define the optimum sintering conditions for the material, several samples were sintered under different conditions (temperature and time were the variables). Density and grain size calculations were used to evaluate each specimen, as well as microstructural appearance.

EXPERIMENTAL PROCEDURE

Powder Lot 1 was examined in the scanning electron microscope (SEM), at various magnifications, to observe the size and shape of the coated powder. A digital X-ray dot map was also constructed to determine the distribution of the nickel and iron coating.

Three different methods of determining the composition of the matrix phase of the three powder lots were used. First, Ultramet reported the results of their energy dispersive analytical X-ray (EDX) analysis as the powder was provided. Secondly, samples from both Lots 1 and 2 were sent to Luvak, Inc., Boylston, MA to determine their composition. There was not sufficient time to send out the third lot; therefore, in addition to this analysis, a third test was performed at the U. S. Army Materials Technology Laboratory (MTL) on all three samples to get a quick analysis of the total matrix content.

Lots 1 and 2 were cold isostatically pressed (CIP) at Industrial Materials Technology, Inc. (IMT), Andover, MA. The samples were sintered in the H_2 atmosphere according to the test matrices shown in Tables 1 and 2. Lot 2 was subjected to both solid state and liquid phase sintering while Lots 1 and 3 were liquid phase sintered only. The majority of the results presented here are from Lot 2. The samples were then examined using an optical microscope and grain size and density calculations were performed. Work proceeded as outlined in the nine tasks listed below.

1. *Coated Tungsten Powder for Ordnance Devices*, Phase I, Small Business Innovative Research Contract No. DAAL02-86-C-0012, Ultramet, Inc., Pacoima, CA, 1986.
2. *Coated Tungsten Powder for Ordnance Applications*, Phase II, Small Business Innovative Research Contract No. DAAL04-88-C-0030, Ultramet, Inc., Pacoima, CA, 1988.

Table 1. ULTRAMET SINTERING MATRIX

Temperature	1200	1250	1300	1350	1400	1450	1500	1550
Time								
1/2					X	X	X	X
1				X	X	X	X	X
2	X	X	X	X	X	X	X	X
4	X	X	X	X				
8	X	X						

Sintering matrix for Powder Lot 2. All temperatures are in degrees Celsius, and times are in hours.

Table 2. ULTRAMET COATED TUNGSTEN POWDER LIQUID PHASE SINTERING STUDY POWDER LOT 1

Temperature (Celsius)	Time (Hours)	Density (g/cc)	Shrinkage (%)
1,500	1	15.71	7.7
1,525	1	16.11	6.8
1,500	2	15.64	7.2
1,550	1	15.82	7.2
1,600	1	15.81	7.2

Sintering matrix for powder Lot 1. All temperatures are in degrees Celsius, and times are in hours.

Task 1: Microscopic Examination

Examination of the coated tungsten powder in the SEM with an analytical X-ray chemical analysis attachment.

Task 2: Chemical Analysis

Chemical analysis was performed at Luvak, Inc. by DC plasma emission on Powder Lots 1 and 2 to determine their compositions.

Task 3: Chemical Analysis by Etching

The amount of matrix present in Powder Lot 3 was calculated by an etching technique that removed the nickel and iron. This was done at MTL, and Lots 1 and 2 were also examined to serve as controls since their compositions were known. All three powders were treated with a solution of 10% nitric acid in methanol. A known weight of coated powder was stirred for one hour at room temperature. The amount of the solution used was much greater than the volume of the powder; approximately 300 ml to 100 g of powder. The powder that remained was washed with distilled water and dried overnight at approximately 150°C. The amount of the matrix phase removed was calculated from the remaining weight of the tungsten. The solutions for this technique were recommended by Brian Williams of Ultramet; he also reported that etching of the tungsten by this process was minimal.[3]

3 Private Communication with Brian Williams, Ultramet, Inc., Pacoima, CA, August 1988.

Task 4: Particle Size Distribution

Particle size distribution of Lot 1 was determined by Microtrac at Dirats Laboratories, Westfield, MA.

Task 5: Cold Isostatic Pressing

Powders from Lots 1 and 2 were CIPed at 25 ksi by Industrial Materials Technology for sintering. Samples from Lot 3 were uniaxially pressed at 24 ksi.

Task 6: Sintering

Button-shaped samples approximately 5/8 inch diameter by 3/8 inch thick were sliced from the CIP bars and were sintered under various conditions of time and temperature in a hydrogen atmosphere furnace. Both liquid phase and solid state sintering was performed. The liquidus for a W, Ni, and Fe alloys of these compositions is approximately 1435°C.[4,5] The sintering cycle included a presintering, oxide reduction treatment. This was done because previous work with tungsten alloys has indicated that oxide reduction is necessary for optimum properties;[6,7] therefore, one hour at 800°C was the treatment used.

Task 7: Density

The density was determined by following ASTM B 328-73, "Standard Test Method for Density and Interconnected Porosity of Sintered Powder Metal Structural Parts and Oil-Impregnated Bearings."

Task 8: Metallography

The sintered samples were sectioned, mounted, and polished; optical micrographs were taken using standard procedures.

Task 9: Grain Size

Grain size was determined according to the intersection method of ASTM E 112-85, "Determining the Average Grain Size."

RESULTS AND DISCUSSION

Material Characteristics

Figures 1a and 1b show the coated tungsten powder of Lot 1. It is seen to be approximately equiaxed to spherical in shape and approximately 15 micrometers in diameter. There is some evidence of the coating on at least some of the particles, particularly at higher magnification. Figure 2 is a digital X-ray dot map showing the distribution of tungsten, nickel, and iron. The map is constructed by determining the origin of the characteristic X-rays that

4. RAYNOR, G. V., and RIVLIN, V. G. *Critical Evaluation of Constitutions of Certain Ternary Alloys Containing Iron, Tungsten, and a Third Metal.* International Metals Review, v. 26, no. 4, 1981, p. 213-249.
5. RAYNOR, G. V., and RIVLIN, V. G. *Addendum To: Critical Evaluation of Constitutions of Certain Ternary Alloys Containing Iron, Tungsten, and a Third Metal.* International Metals Review, v. 28, no. 2, 1983, p. 122-129.
6. RABIN, B. H., BOSE, A., and GERMAN, R. M. *Characteristics of Liquid Phase Sintered Tungsten Heavy Alloys.* International Journal of Powder Metallurgy, v. 25, no. 1, p. 21.
7. BOSE, A., and GERMAN, R. M. *Sintering Atmosphere Effects on Tensile Properties of Heavy Alloys.* Met. Trans. A, v. 19A, October 1988, p. 2467.

result from the interaction of the electron beam with the sample. The tungsten map is a representation of the powder particle substrates, while the nickel and iron signals reveal the distribution of the coating on the tungsten. It is noted here that the coating is quite uniform.

The compositions of the three powders calculated by all three methods are shown in Figure 3. The compositions, determined by DC plasma emission, of the matrix phase of Lots 1 and 2 were much less than those reported by Ultramet using an EDX attachment on the SEM. The EDX procedure greatly overestimated (by 10 to 15 times) the true amount of coating present. For this reason Ultramet concluded, erroneously, they were producing powder with the targeted amount of matrix phase (about 5 weight-percent combined nickel and iron). The MTL matrix etching technique results correlated closely with the chemical analysis from Luvak. The reported value from Ultramet for Lot 3 was not taken directly from an EDX run, but rather from comparison of EDX results to another powder with known results from both EDX and independent chemical analysis.[3] Since the etching test results from Lots 1 and 2 are close to the actual values, it is assumed this value from MTL is a fairly good approximation.

Typically, satisfactory sintering is expected from an alloy of 95% W with a matrix of nickel and iron in a ratio of 7:3, and is shown in Figure 4. The amount of the matrix on the coated tungsten particles increased with each successive batch and, consequently, yielded better sintering results. The corresponding change in the theoretical density is given in Table 3. The lack of nickel and iron, however, may not be the only hindrance to full densification.

Table 3 THEORETICAL DENSITIES OF ULTRAMET POWDERS

Lot No. 1	19.11 g/cc
Lot No. 2	18.75 g/cc
Lot No. 3	18.25 g/cc

The results from the particle size determination are displayed in Table 4 and Figures 5 and 6. Figure 5 shows an average particle size of 18.44 micrometers. Figure 6 is a histogram showing the Microtrac results. The particles are extremely narrowly distributed, as displayed, and if the fact that Microtrac gives erroneous values at both ends of the distribution is taken into consideration, then the actual size distribution is further narrowed.[8] A monosized particle cannot be as densely packed as bimodally sized particles.[9] This, however, may not be such a simple problem to solve since the CVD technique being used by Ultramet, which insures an even coating on the particles, requires a rather restricted particle size distribution to obtain good fluidization of the powder in the CVD reactor.[10]

Sintering Properties

The density and shrinkage results of liquid phase sintered Lot 1 samples are displayed in Table 2. The method used to determine the densities of these samples, ASTM B 311-86, "Density of Cemented Carbides," was not the same as the method used for the other lots. Using this standard to measure the density of a part which is not fully dense yields inaccurate

8. Powder Injection Molding Symposium. RPI, July 16-18, 1990.
9. GERMAN, R. M. *Powder Injection Molding*. Metal Powder Industries Federation, Princeton, NJ, 1990, p. 23-56.
10. Private Communication with Jack Stiglich, Ultramet, Inc., August 16, 1990.

results. A false low reading of the sample weight suspended in water is given because of the air entrapped in the sample's pores and, therefore, a false high density calculation results. These results, although probably falsely high, are only about 82% to 84% of the theoretical density.

Table 4. PARTICLE SIZE DISTRIBUTION BY MICROTRAC

Fraction	Cum % W
Finer than 62 micron	100.0
Finer than 44 micron	91.2
Finer than 31 micron	92.8
Finer than 22 micron	72.7
Finer than 16 micron	43.9
Finer than 11 micron	21.4
Finer than 7.8 micron	9.1
Finer than 5.5 micron	3.9
Finer than 3.9 micron	1.1
Finer than 2.8 micron	0.1

Average 18.44 micron.

The density results of the samples from Lot 2 sintered according to the matrix in Table 1 are presented in Figures 7 and 8. The same data is presented in both two-dimensional and three-dimensional formats. The trend shown by the data is that the density increases with increasing temperature, but time does not appear to have much effect. It is important to notice that the densities are only approaching 90% of the theoretical value. Not much densification is occurring in the solid state sintering regime. Only when the matrix melts does the density rise to any significant degree.

The grain size data also shows a transition point between solid and liquid phase sintering, as shown in Figure 9. The tungsten grain size is fairly constant up to 1400°C; beyond that, the grain size of the samples rapidly increased. This is a characteristic of liquid phase sintering. It is not, however, always desirable because of the loss of strength associated with coarser grains (e.g., Hall-Petch[11]). Note that the liquid matrix material promotes the growth of tungsten grains.

Figures 10a and 10b show the difference in the appearance of the microstructure between Lots 2 and 3. While both structures contain a lot of porosity, more matrix is noticeable in the Lot 3 micrograph. This agrees with the reported and calculated compositions. The grains in Lot 3, however, appear slightly larger than those in Lot 2; this is shown by the grain size measurements displayed in Figure 11. With the assumption that the particles from both Lots 2 and 3 were originally the same size, the figure illustrates the tungsten grain growth is promoted by the liquid matrix phase.

11. DIETER, G. E. *Mechanical Metallurgy*. 2nd Ed., McGraw Hill, New York, NY, 1976.

The change in microstructure with respect to temperature can be observed in Figures 12a through 12f. The tungsten grains of the solid state sintered material, below 1435°C, were polycrystalline. The samples sintered at liquid phase lost this feature and became single crystal particles surrounded by either matrix material and/or pores. Both grain growth and densification can be observed by examining these microstructures in order of increasing temperature.

Another interesting occurrence was observed macroscopically on the polished samples. The liquid phase sintered samples were not uniform in their density. The region at the bottom of the sample was more dense than the top and outer edges. Influenced by gravity, the matrix phase melted and flowed to the bottom of the sample. One would expect capillarity to have a greater influence in filling the pores, but the pores of this rather large, monosized powder are too big to have much effect.

RECENT WORK AND FUTURE PLANS

In an effort to investigate the possibility of more satisfactory results from a bimodally-sized powder, a new powder blend was formulated and mixed at MTL and sintered at 1500°C for one hour in hydrogen. The composition of this powder was:

- 80% Ultramet Lot 3 (coarse particles)
- 20% 95% W, 3.5% Ni, and 1.5% Fe (fine particles)

The fine powder is estimated to be about 0.5 to 1 micron in size, while Powder Lot 3 is assumed to be about the same size as Lot 1 which had the particle size analysis (approximately 18 microns).

The density of this sample was calculated, by the oil infiltration technique, to be 93% of the theoretical density. This is the best density result out of all the sintered samples, indicating that the narrow particle size distribution is a significant negative factor in the densification process.

Further work will examine the effect of various size mixtures investigating the size and amount of powder added. Initially bimodal blends but, as experience grows, trimodal and continuous distributions will be examined.

CONCLUSIONS

It was possible to sinter these three Ultramet powders only to approximately 90% density. Temperatures above the liquidus produced the best results. In this temperature region grain growth is enhanced by the presence of the liquid matrix, but there was insufficient matrix to fill the pores between the tungsten grains to approach full density. Another possible explanation for the poor densification may be that the powder had narrow distribution of coarse particles, essentially a monosized powder. A wider distribution of particles, although not favorable to the CVD processing technique, may produce better sintered densities. This possibility is currently under consideration and initial results indicate that the bimodal powder will have the better sinterability.

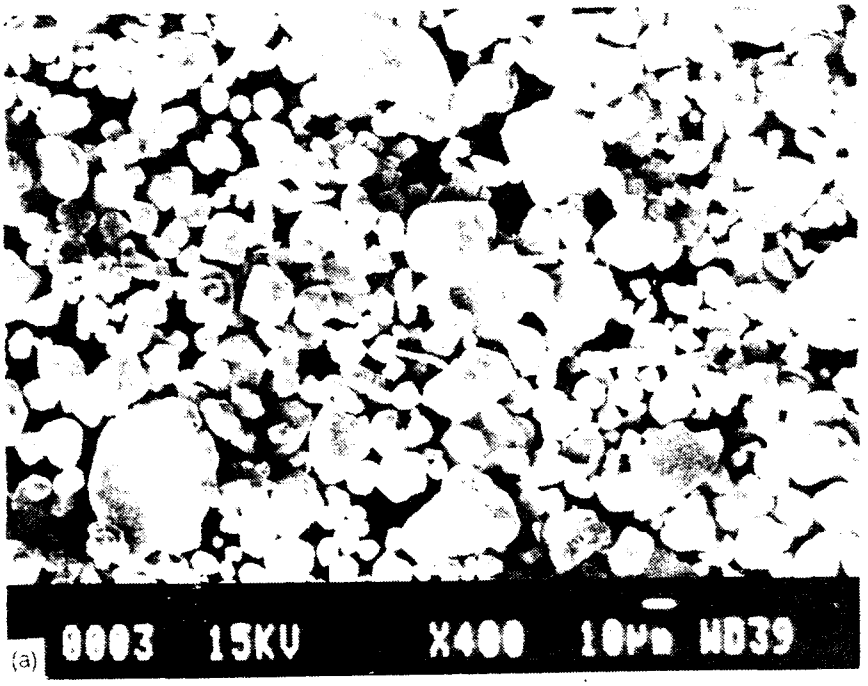
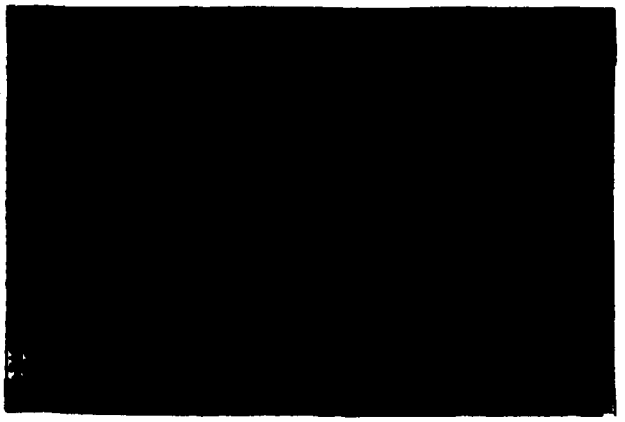
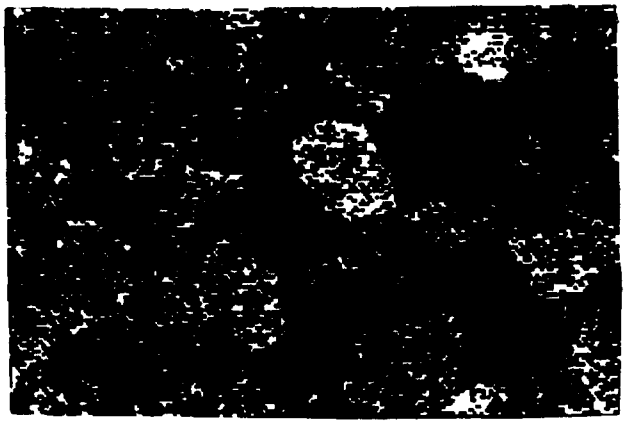


Figure 1 SEM of Ultramet Powder Lot 1



TUNGSTEN



NICKEL

IRON

Figure 2. Digital X-ray dot map of Lot 1 showing the location of the tungsten particles and the nickel and iron coatings. Note that the nickel and iron signals are uniformly dispersed on the tungsten powder particles.

Composition of Ultramet Powders
(Balance is Tungsten)

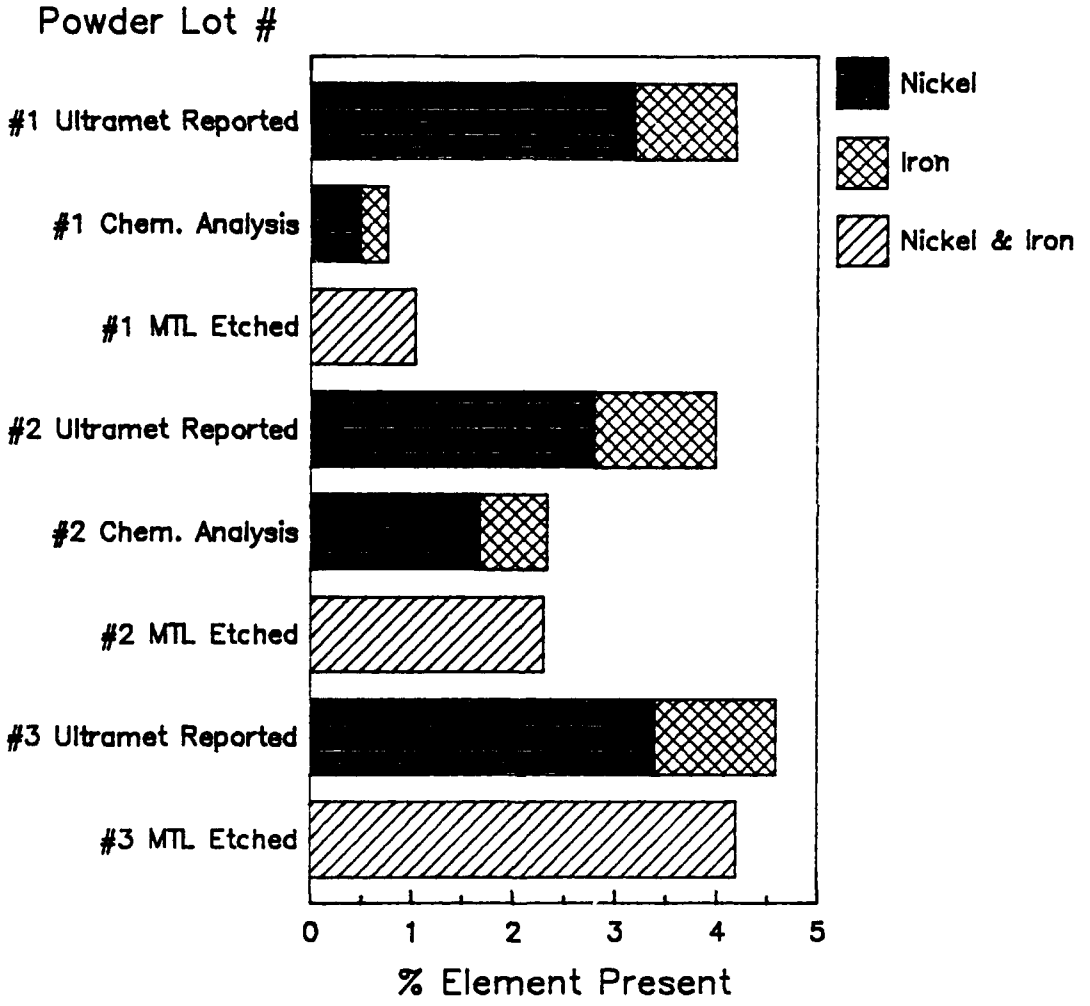


Figure 3. Composition of the nickel and iron coating for Powder Lots 1 through 3. The methods of each analysis can be found in the test.

Mag. 500X

8μ

Figure 4. Microstructure of typical 95% W, 3.5% Ni, and 1.5% Fe heavy alloy.

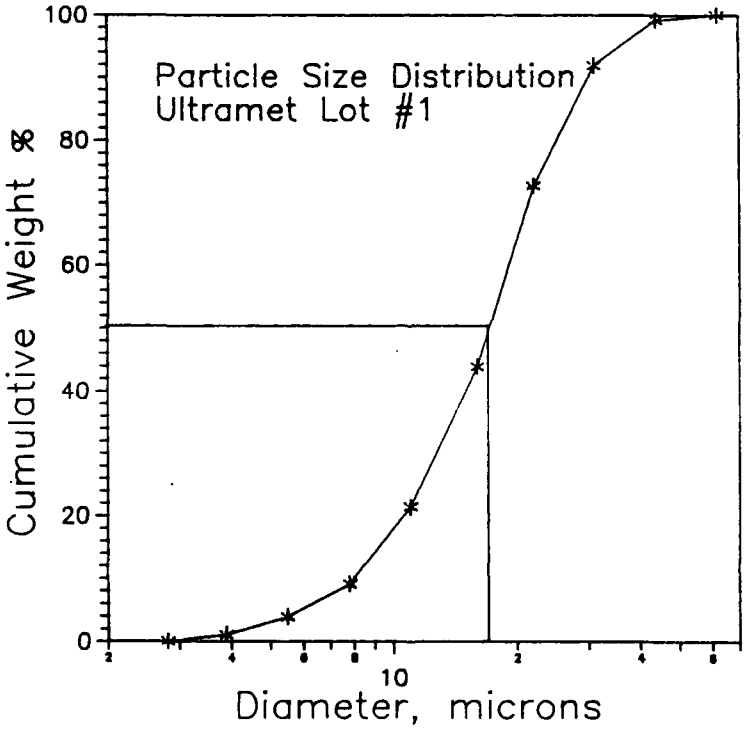


Figure 5. Particle size distribution of Powder Lot 1.
Results from Microtrac particle size analysis.

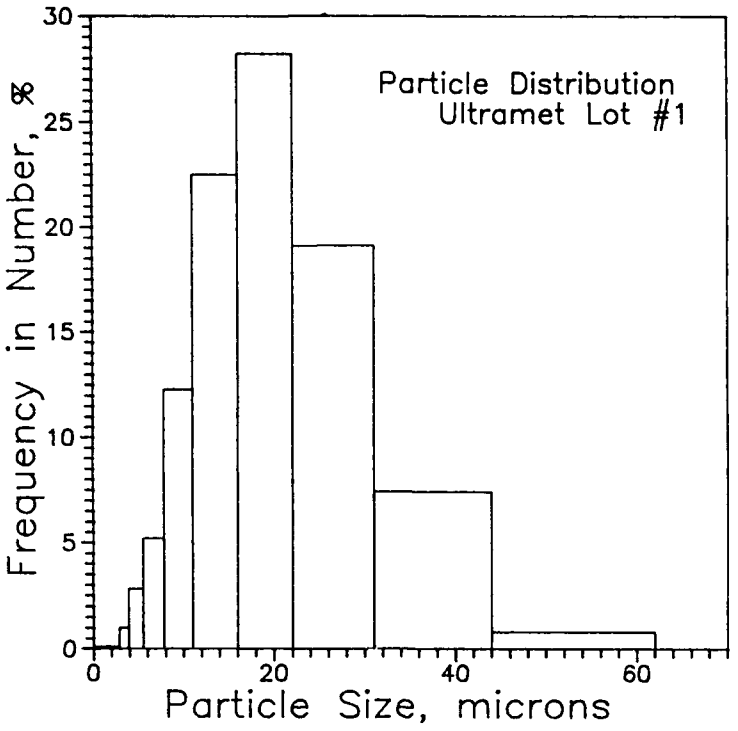


Figure 6. Particle size distribution of Powder Lot. 1. Results from Microtrac particle size analysis, displayed as a histogram.

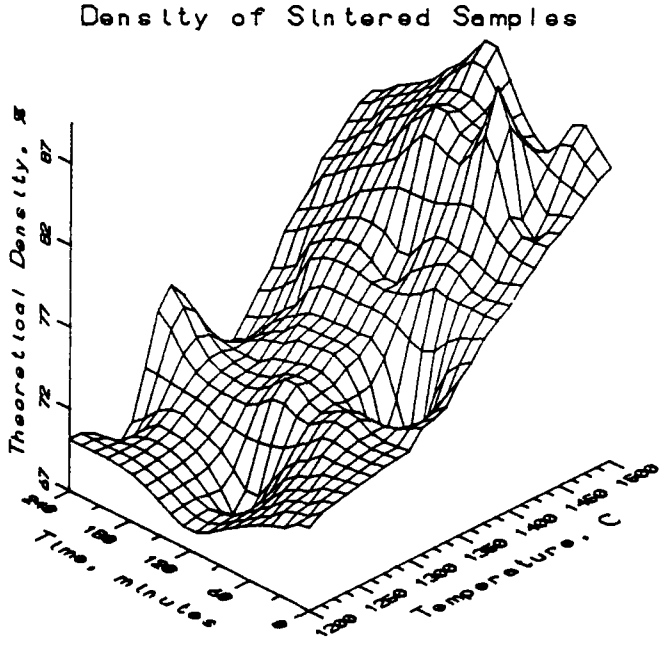


Figure 7. Three-dimensional plot of the sintering results of Powder Lot 2. Temperature is in degrees Centigrade, time is in minutes, and theoretical density is shown as a percent of the calculated density for Lot 2 (18.75 g/cc).

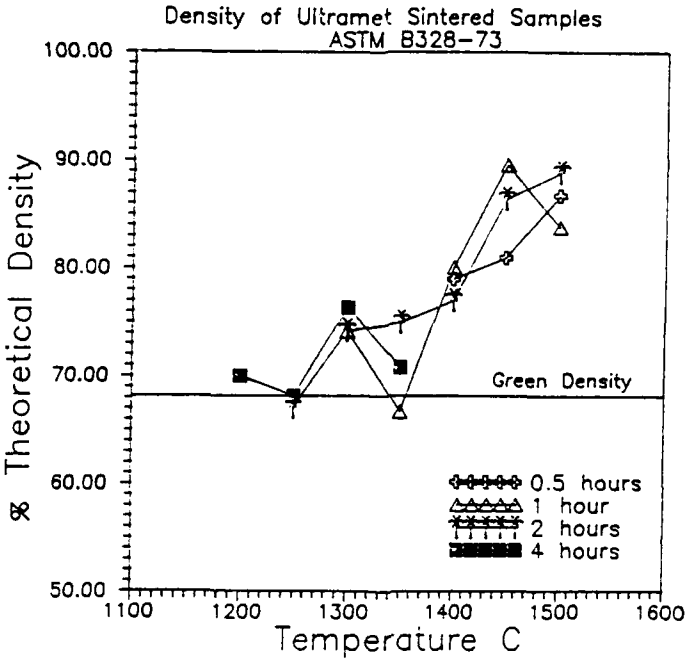


Figure 8. Two-dimensional plot of the sintering results of Powder Lot 2. Temperature is in degrees Centigrade, time is in minutes, and theoretical density is shown as a percent of the calculated density for Lot 2 (18.75 g/cc). Green density is shown at 68%; this is the density of the CIP powder.

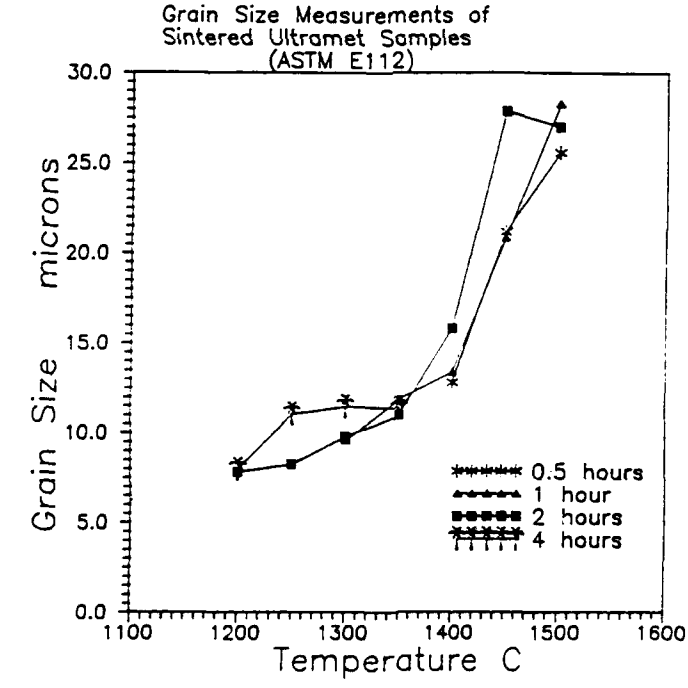
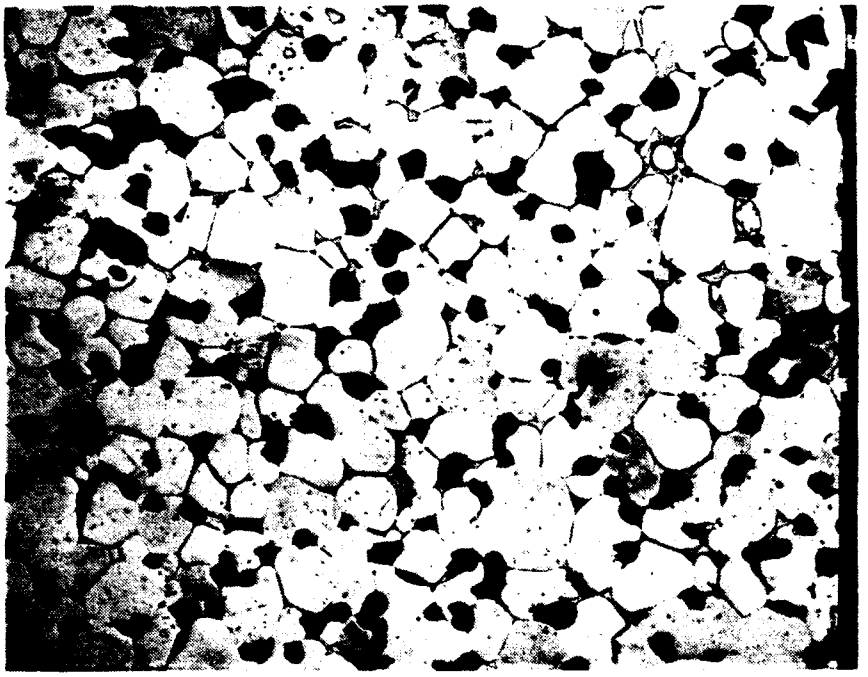


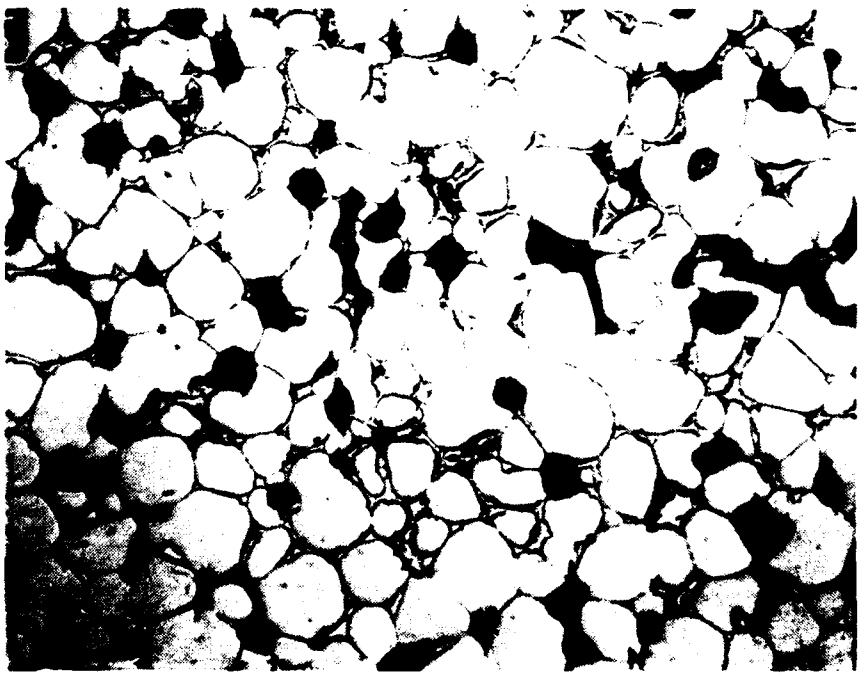
Figure 9. Plot of tungsten grain size versus sintering temperature for Powder Lot 2. Temperature is in degrees Centigrade, and the grain size is in micrometers.



Mag. 200X

20 μ

Figure 10a. Photomicrographs of sintered samples from Lot 2.
Samples sintered at 1500°C for one hour.



Mag. 200X

20 μ

Figure 10b. Photomicrographs of sintered samples from Lot 3.
Samples sintered at 1500°C for one hour.

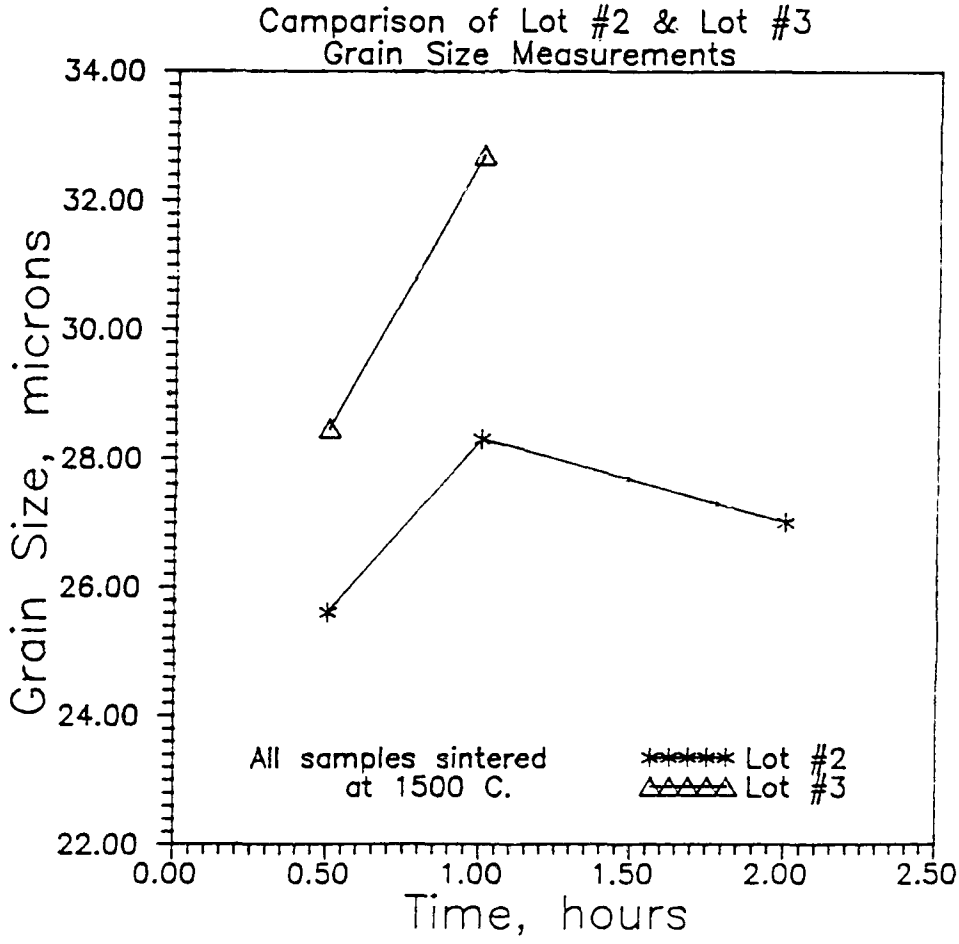
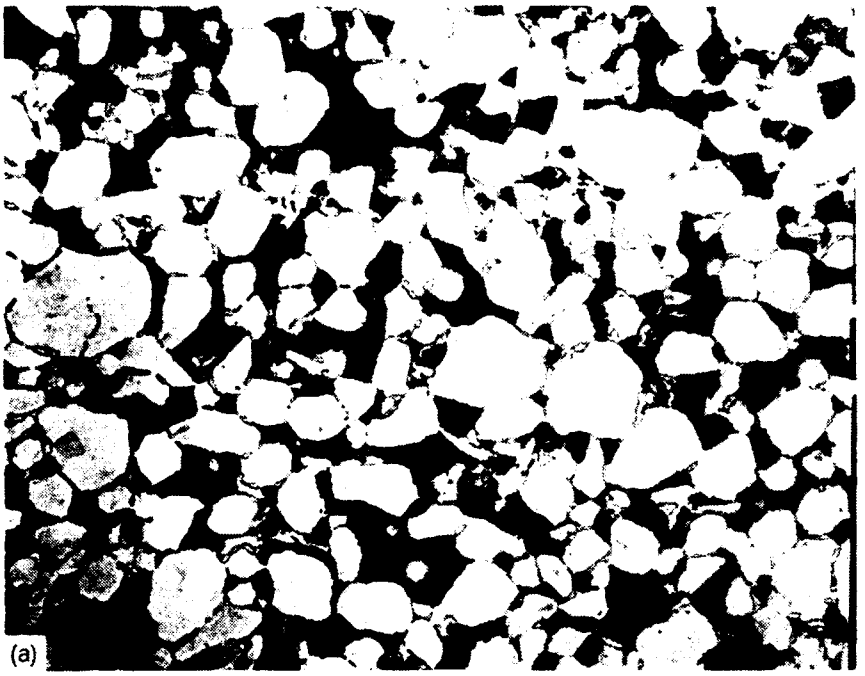


Figure 11. Comparison of the grain size from Powder Lots 2 and 3 for various sintering times at 1500°C. Grain size in micrometers.



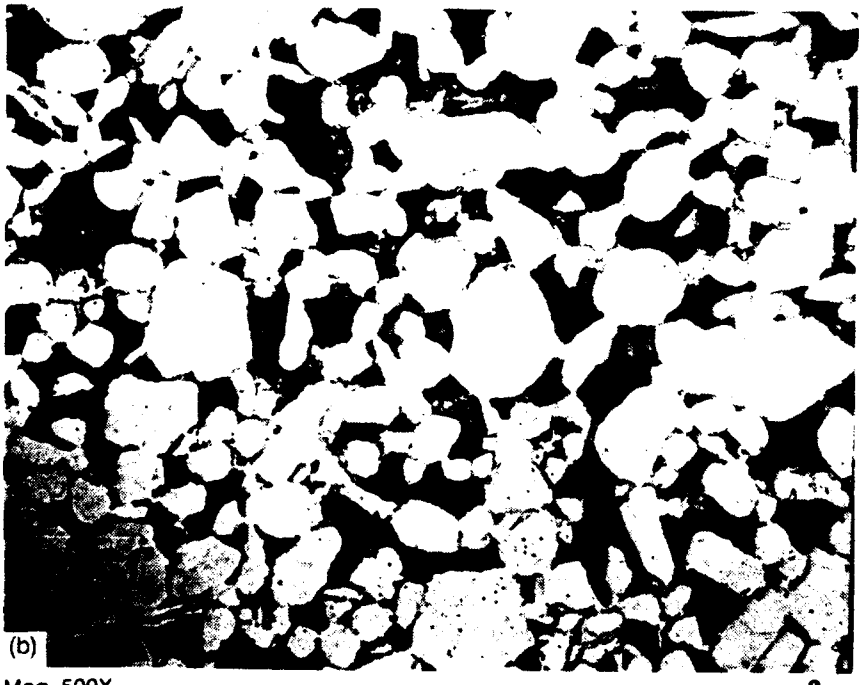


Figure 12. Isochronal (two hours) sintering of Powder Lot 2 from 1200°C to 1500°C. Temperatures below 1435°C are solid state sintered.

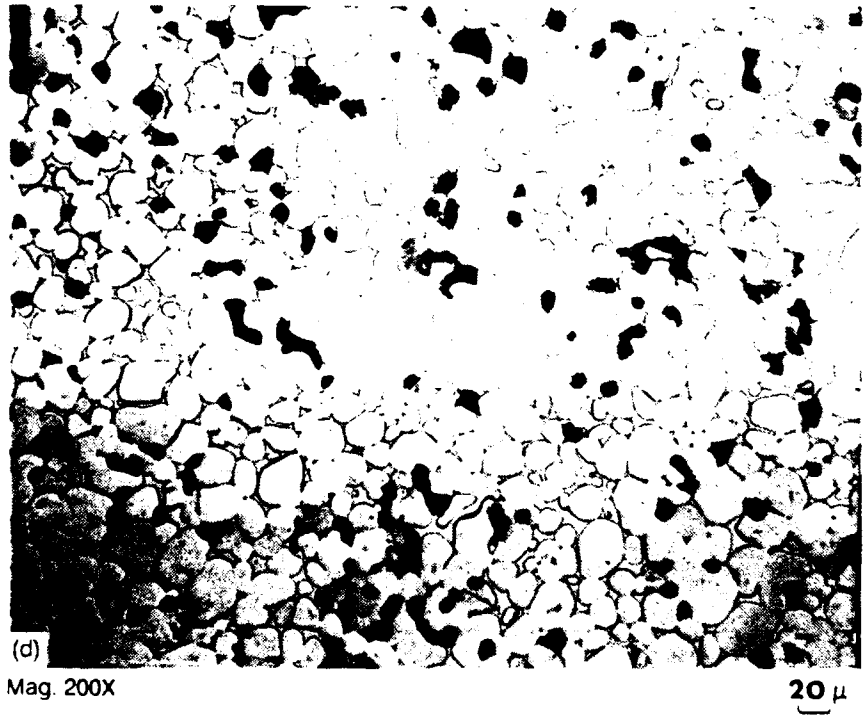
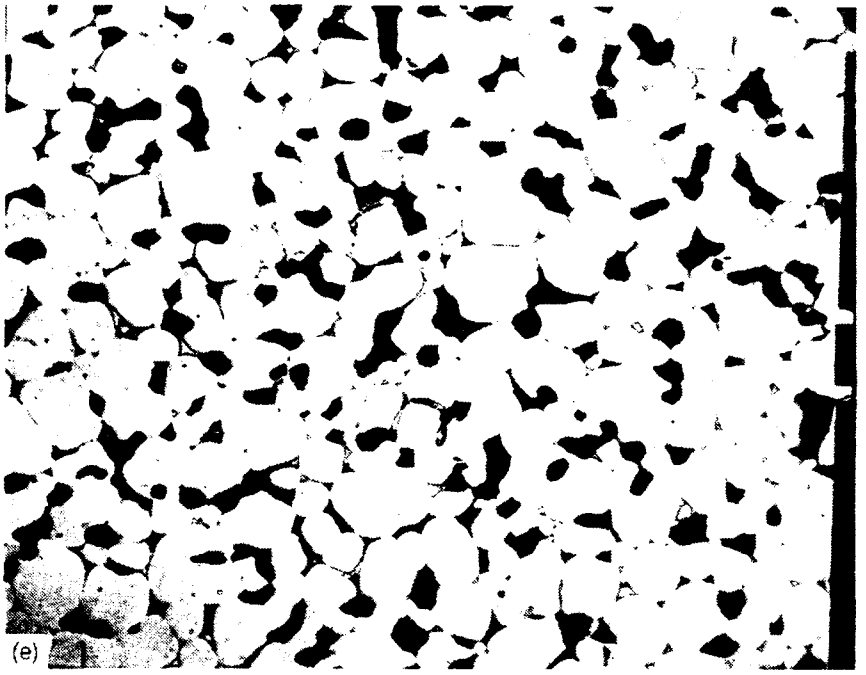


Figure 12 (cont'd). Isochronal (two hours) sintering of Powder Lot 2 from 1200°C to 1500°C. Temperatures below 1435°C are solid state sintered



Mag. 200X

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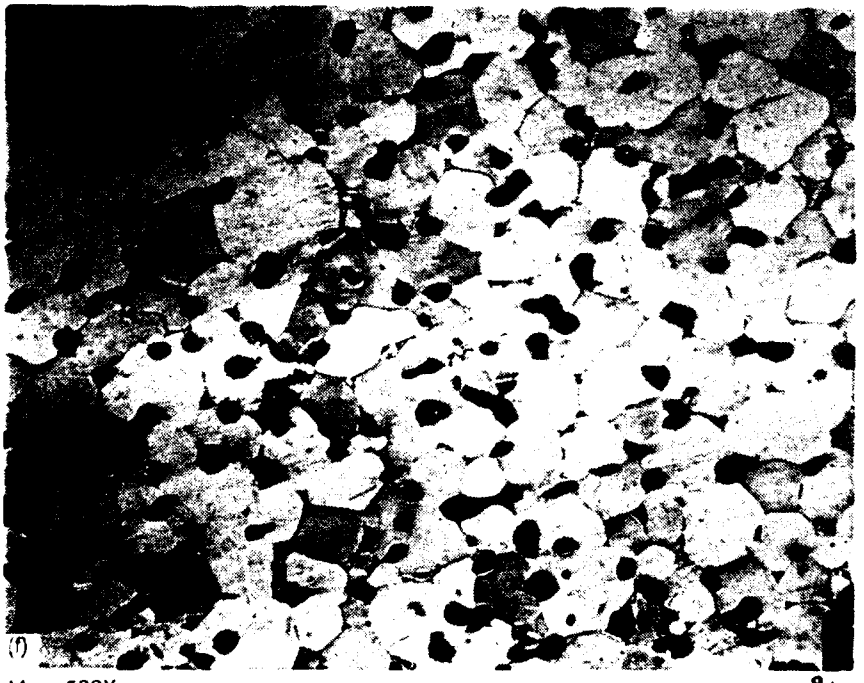

Mag 500X

8 μ

Figure 12 (cont'd) Isochronal (two hours) sintering of Powder Lot 2 from 1200°C to 1500°C. Temperatures below 1435°C are solid state sintered

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SINTERABILITY OF TUNGSTEN POWDER CVD COATED WITH NICKEL AND IRON -
Sharon Mulligan and Robert J. Dowding

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Key Words

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Powder metals
Tungsten powders
Chemical vapor deposition (CVD)

Three lots of tungsten powder coated with nickel and iron by chemical vapor deposition (CVD) were characterized as to its composition, particle size, and size distribution. This was accomplished through the use of the scanning electron microscope (SEM) with a digital X-ray dot mapping attachment, DC plasma emission, and Microtrac analysis. The sinterability of the powder was also evaluated through sintering trails at temperatures above and below the liquidus of the matrix phase. It was found that the coating of nickel and iron on the tungsten substrate was very uniform, but it was also difficult to obtain high sintered densities due to the near monosized nature of the powder. The highest sintered density obtained was 90% of the theoretical value. It was concluded that to obtain full density a wider distribution of the particle size would be needed